National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1021

Glass Beads - Particle Size Distribution

This Standard Reference Material (SRM) is intended for use in the evaluation and calibration of equipment used to measure particle size distributions (PSD) in the 2 μm to 12 μm diameter range. Typical methods for PSD determination would be laser light scattering (LLS), electrical sensing zone (ESZ), and sedimentation. The size range of this SRM follows that of the coarser beads of SRM 1003c. Each unit of SRM 1021 consists of a single bottle containing approximately 4 g of solid spherical soda-lime glass beads.

Certified Values: The certified cumulative volume (mass) distribution was determined using both electric sensing zone and laser light scattering techniques. The certified values and associated uncertainties based on the average of results from the two techniques are given in Table 1.

Information Values: Information values based on ESZ and LLS techniques are provided in Table 2 [1]. A comparison of the ESZ and LLS techniques is shown in Figure 1.

Expiration of Certification: The SRM bottle must be kept tightly capped with a desiccator pack inside. This is to prevent moisture absorption that can cause clumping of the powder. The certification of this SRM is valid until **31 January 2009**, within the measurement uncertainties specified, provided the SRM is used in accordance with the instructions given in this certificate.

Certification Procedure: Two splits from each of ten bottles were analyzed with both the ESZ and LLS techniques to measure the diameter at which a specified percentage of the material is smaller. The percentages are 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 70, 75, 80, 85, and 90. For each percentage, the only experimental setting that changed between the observations was the bottle of material.

The SRM preparation, measurement, and certification were performed by J.F. Kelly of the NIST Ceramics Division.

Statistical analyses were performed A.I. Avilés of the NIST Statistical Engineering Division.

The support aspects of the preparation, certification and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Service Division.

Gabrielle Long, Acting Chief
Ceramics Division

Gaithersburg, MD 20899
Certificate Issue Date: 27 March 2003

John Rumble, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

The sample preparation procedures described here for the certification measurements should be followed to obtain representative sampling and particle dispersion. The LLS and ESZ techniques require much less material than the 4 g provided and special care must be exercised when taking subsamples from the SRM bottle. Representative subsamples of this SRM can be obtained by holding the bottle horizontal and rotating several times before using a spatula to extract about 20 mg powder. Repeat this procedure twice more to extract a total of about 60 mg. Add the powder to 20 mL of an appropriate fluid in a cuvet. The cuvet is capped and shaken to disperse the particles, then placed in an ultrasonic bath for 35 minutes to break up any agglomerates. Before sampling, the cuvet is inverted several times to mix the particles in the fluid. Aliquots of approximately 1 mL are taken by pipette to inject into the measuring system. Add sufficient aliquots until the instrument indicates that a sufficient amount has been added to produce an acceptable concentration.

ELECTRICAL SENSING ZONE METHOD

The ESZ method for determining powder size distribution uses particles suspended in a conductive fluid such as saline solution. The suspension is drawn through an orifice or aperture separating two electrodes, between which, an electric potential is applied. A particle passing through the orifice (sensing zone) causes a pulse in the circuit impedance, which is proportional to the particle volume. The measured impedance change is converted to a particle volume using a calibration constant obtained by measuring particles of known volume. For a given aperture of diameter D, the working range is from 0.02D to 0.60D. For SRM 1021, an instrument with a 50 μm aperture was used to enable measurements over the range from 1 μm to 30 μm. The conductive fluid was an aqueous solution of 0.8 % mass fraction NaCl. The size distributions measured for the ten bottles of SRM 1021, selected by stratified random sampling, are given in Table 2.

LASER LIGHT SCATTERING METHOD

The LLS analysis used filtered deionized water to suspend the glass beads. Aliquots of approximately 1 mL were taken by pipette to inject into the measuring system. The LLS technique uses approximations to the Mie scattering theory [2] to convert the measured scattering pattern to particle size distribution. Mie theory includes the influence of diffraction, refraction, reflection and polarization effects. This theory requires that we know the real and imaginary refractive indices of the particles and the suspending medium and that the particles are optically homogeneous smooth spheres. The use of glass spheres in a transparent medium is a nearly ideal design to satisfy the Mie assumptions. A refractive index of 1.52, with an imaginary component of zero, was used for the soda lime glass. The size distributions measured by LLS, for the same ten bottles measured by ESZ, are given in Table 2. A graphical comparison of the mean distribution measured by LLS with the mean distribution obtained by ESZ is shown in Figure 1.

Certified Values and Uncertainty Determination: The certified values and uncertainty estimates are shown in Table 1. The lme function from the nlme library for the language S was used to fit the linear mixed-effects model using the *restricted maximum likelihood* (REML) estimation method [3,4]. The error analyses for the certified diameter values provided with the SRMs follow recommendations contained in the ISO Guide [5] and in NIST Technical Note 1297 [6]. Three sources of error were evaluated in determining the uncertainty values: 1) method accuracy, 2) reproducibility of the measurements, and 3) bottle to bottle differences in the material. The first of these is a Type B uncertainty obtained by analysis of the calibration results using SRM 1961, while the latter two are obtained by the REML analysis. These uncertainty terms are combined using a root sum of squares to obtain a combined standard uncertainty. Since there are sufficient data (>30) a normal distribution is assumed and thus the expanded (95 %) uncertainty values can be obtained by multiplying the combined standard uncertainty by $k = 2$.

Table 1. Certified Diameter Values

Cumulative Volume Fraction Finer* (%)	Certified Diameter (μm)	Standard Uncertainty Homogeneity (μm)	Standard Uncertainty Measurement (μm)	Type B Standard Uncertainty (μm)	Combined Standard Uncertainty (μm)	Expanded** Uncertainty (μm)
5	2.1	0.01	0.07	0.25	0.26	0.52
10	2.6	0.00	0.09	0.25	0.27	0.53
15	3.0	0.00	0.10	0.25	0.27	0.54
20	3.3	0.00	0.11	0.25	0.27	0.55
25	3.7	0.00	0.12	0.25	0.28	0.55
30	4.1	0.00	0.13	0.25	0.28	0.56
35	4.5	0.00	0.13	0.25	0.28	0.56
40	4.9	0.00	0.12	0.25	0.28	0.56
45	5.3	0.00	0.12	0.25	0.28	0.55
50	5.8	0.00	0.11	0.25	0.27	0.55
55	6.3	0.00	0.11	0.25	0.27	0.54
60	6.8	0.00	0.10	0.25	0.27	0.54
65	7.4	0.00	0.12	0.25	0.28	0.56
70	8.1	0.00	0.15	0.25	0.29	0.58
75	8.9	0.00	0.20	0.25	0.32	0.63
80	9.9	0.00	0.26	0.25	0.36	0.72
85	11.1	0.00	0.35	0.25	0.43	0.86
90	12.9	0.00	0.51	0.25	0.57	1.14

* The cumulative volume fraction finer is the portion of SRM 1021 smaller than the certified diameter value.

**The uncertainty at each percentile, computed according to the ISO and NIST Guides [5,6], is an expanded uncertainty at the 95 % level of confidence.

Table 2. Information Diameter Values (Ten Bottle Averages) Measured by LLS and ESZ

Cumulative Volume Fraction Finer* (%)	ESZ (μm)	LLS (μm)
5	2.11	2.05
10	2.62	2.50
15	3.04	2.89
20	3.43	3.26
25	3.82	3.63
30	4.21	4.00
35	4.60	4.39
40	5.00	4.80
45	5.41	5.23
50	5.85	5.69
55	6.32	6.20
60	6.83	6.75
65	7.39	7.38
70	8.04	8.09
75	8.81	8.93
80	9.75	9.95
85	10.97	11.26
90	12.77	13.11

*The cumulative volume fraction finer is the portion of SRM 1021 smaller than the stated diameter.

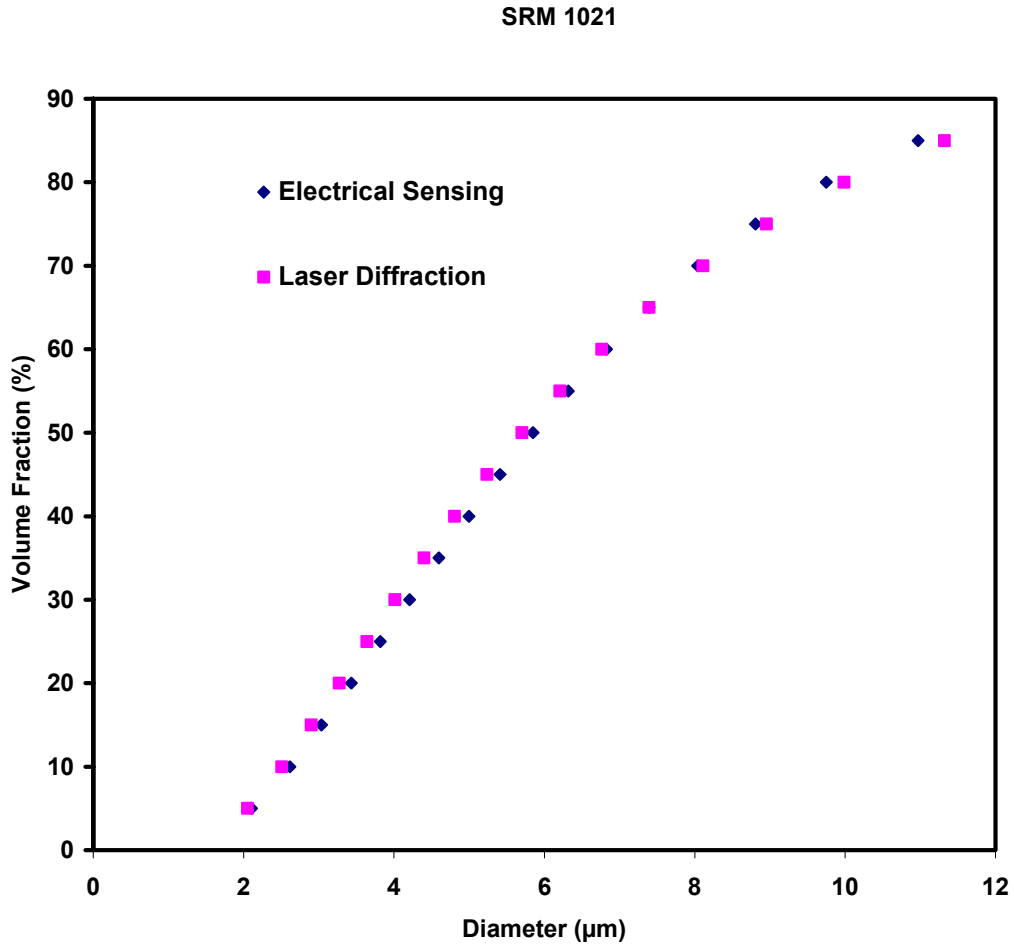

Figure 1. Comparison of the ESZ and LLS results for SRM 1021

REFERENCES

[1] May, W.E.; Parris, R.M.; Beck II; C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurement*, NIST Special Publication 260-136 (2000).

[2] Bohren, C.F.; Huffman, D.R.; Absorption and Scattering of Light by Small Particles, John Wiley, New York (1983).

[3] Searle, S.R.; Casella, G.; McCulloch, C.E.; Variance Components, New York: John Wiley (1992).

[4] Pinheiro, J.C.; Bates, D.M.; Mixed-Effects Models in S and S-PLUS, New York: Springer (2000).

[5] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

[6] Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Estimating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.